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(54) MAGNETIC RECORDING MEDIUM

(57)Abstract:
PURPOSE: To provide an ultrahigh density magnetic recording medium having high reproduction output and satisfactory electromagnetic transducing characteristics such as overwriting characteristicshardly undergoing electrification and excellent in running durability.
CONSTITUTION: When a nonmagnetic layer based on nonmagnetic powder and a resin binder and a magnetic layer based on ferromagnetic powder and a resin binder are successively formed on a nonmagnetic substrate to obtain a magnetic recording mediumthe magnetic layer is composed of at least an upper layer and a lower layerthe average particle diameter of the ferromagnetic powder in the lower layer is made larger than that in the upper layer and the total thickness of the magnetic layer is regulated to ≤0.8μm. The nonmagnetic layer and the magnetic layer are preferably formed by a wet- on-wet coating systemferromagnetic metal powder or ferromagnetic hexagonal ferrite powder is preferably used as the ferromagnetic powder and at least carbon black is preferably used as the nonmagnetic powder.

CLAIMS

[Claim(s)]
[Claim 1]In a magnetic recording medium with which it comes to form a magnetic layer which makes a subject a non-magnetic layerferromagnetic powderand bonding agent resin which make a subject nonmagnetic powder and bonding agent resin in this order on a nonmagnetic substrateA magnetic recording mediumwherein it consists of two-layer at leastmean particle diameter of said lower layer ferromagnetic powder is larger than mean particle diameter of ferromagnetic powder of said upper layer and overall thickness of this magnetic layer is [of the upper layer and a lower layer] 0.8 micrometer or less. [magnetic layer / said]
[Claim 2]The magnetic recording medium according to claim 1wherein said non-magnetic layer and a magnetic layer are formed with a wet-on-wet coating method.
[Claim 3]The magnetic recording medium according to claim 1 or 2 in which said ferromagnetic powder is ferromagnetic metal powder.
[Claim 4]A magnetic recording medium given in any 1 paragraph of claim 1 in which said ferromagnetic powder is ferromagnetic hexagonal ferrite powder - claim 3.
[Claim 5]A magnetic recording medium given in any 1 paragraph of claim 1 in which said at least some of nonmagnetic powder is carbon black - claim 4.
[Claim 6]A magnetic recording medium given in any 1 paragraph of claim 1 whose mean particle diameter of said carbon black is less than 40mmicro and whose DBP oil absorption is 300 mL(s)/not less than 100g - claim 5.
[Claim 7]A magnetic recording medium given in any 1 paragraph of claim 1 in which said magnetic recording medium is a magnetic recording disk - claim 6.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application]This invention relates to the magnetic recording medium which fitted the magnetic disk for data recording especially about the magnetic recording medium for high density recording.
[0002]

[Description of the Prior Art]The electronization of a signal is easy and construction of the system by combination with circumference electronic equipment is [that the repeated use of a medium is possible for magnetic-recording art] possibleSince it has the outstanding feature which is not in other recording methodslike correction of a signal can also be simplifiedit is broadly used in various fields including videoan audioa computer useetc. And in order to satisfy the demand of long-time-izing of the miniaturization of apparatushigh-definition-izing of a record reproduction signaland recordincrease of storage capacityetc.to a recording mediummuch more improvement in storage density has always been desired. And furthermore the policy which makes grain size of ferromagnetic powder smallraises the dispersibilityor raises the packing degree in the inside of the magnetic layer in the magnetic recording medium of a coating mold is proposed variouslyas an effective meansUsing the ferromagnetic metal powder which was excellent in magnetic propertiesa hexagonal ferriteetc. is also performed.

[0003]In connection with the spread of the minicomputer as OA equipmentand personal computersthe spread of the magnetic recording disks as external storage is remarkableThe frequency in use of a magnetic recording disk spreadsand it is used and saved by a broad environmental condition about temperature humidityand the dust of an operating environment has also come to be used at many places. In order to attain large-scale-izing of recordand a miniaturization especiallyimprovement in storage density is demanded stronglybut in order to obtain the magnetic recording medium which fits high density recording using needlelike ferromagnetic powder like beforeit is necessary to make the upper limit of needlelike ferromagnetic powder smaller enough than a recording wavelength or recording bit length. Nowas needlelike ferromagnetic powderpractical use is already presented with the thing with a size of about 0.3 micrometerand the shortest recording wavelength of about 1 micrometer or less of it has become possible.

[0004]In order to obtain the medium in which future still higher-density record is possibleit is necessary to make the size of needlelike ferromagnetic powder still still smaller. Howeverin such small needlelike ferromagnetic powderSince thickness becomes very thin with 100A or less and becomes very as small also as particle volume as below $10^{-17}cm^3$there are problems -- sufficient orientation is not obtainedeven if magnetic properties fall by the effect of a thermal agitation and the surface and it adds a magnetic field to a magnetic coating film.

[0005]Ferromagnetic metal powder is examined as ferromagnetic powder corresponding to high density recording until nowThe magnetic recording medium using the hexagonal ferrite particles which have an easy axis in the direction vertical to a plate surface by plate-like in recent years as ferromagnetic powder was developed (for exampleJP58-6525A58-6526etc.). By these0.05 micrometer or less of the mean particle diameter of ferromagnetic powder became possibleand high-density-recording-ization of it was attained.

[0006]Furthermorelarge narrow track width has been required for high density recording. Development and utilization are wholeheartedly considered so that to fill these demandsand use of the ferromagnetic metal powder which can expect high power and high recording density also in a magnetic diskor ferromagnetic hexagonal ferrite powder may be considered and it may meet the miniaturization of sizeand the demand of improvement in the storage density of a medium. The lamination of a magnetic layer and high power are desired as improvement in high recording density and an over-writing magnetic parametric performanceand we are especially anxious about running durability remarkable deterioration in connection with lamination.

[0007]Namelyalthough overwrite (over-writing) of the record signal with which magnetic wave length differs is usually required in the magnetic recording medium of computer usessuch as a floppy diskAlthough over-writing of two kinds of signals which have a twice as many relation as this on frequencyl fand 2 f signals should just have been completed conventionallyFrom the magnetic recording disk with a high capacity of 10 M bytes or more demanded strongly these daysover-writing of two or more signals which the recording wavelength not only became shortbut have it in a broadband from that of the frequency ratios 3:8such as a RLL signalis demanded. When a signal with a short recording wavelength and a large difference of recording frequency is usedin order to perform overwrite (over-writing) well on a signal with a long recording wavelengtha signal with a short recording wavelengthThere was a limit only by raising the magnetic properties of a magnetic layer as indicated by JP58-122623AJP61-74137Aetc.

[0008]That isin a magnetic layer with an old thickness of 1.0 micrometers or moresince a line of magnetic force does not reach till the deep place of a magnetic layer even if it carries out overwrite of the short record signal by the record signal top of long wave length rather than recorded previouslyit is recorded previously and the signal of reliance long wave length cannot be eliminated. The gap of a recording head is becoming narrow with improvement in storage density. In connection with thissufficient record for the thick allies of a medium is becoming difficult.

[0009]Thenif the magnetic layer was made thin to 1 micrometer or less in order to solve the above-mentioned problema magnetic layer will exfoliate easilyrunning durabilitysuch as becoming a generation factor of a dropoutcould not be securedbut the problem that reliability fell arose. Thereforeespecially in order to provide the magnetic recording medium which can respond to the aforementioned high-density-recording-izationthe improvement in a reproducing outputreservation of an overwrite characteristicand running durability have posed a big problem.

[0010]Electrification at the time of a run of a magnetic recording medium caused increase of the number of dropouts by adhesion of dustandespecially in the case of the magnetic recording medium for data recordingit became a defect with a fatal error rate by it. In order to improve the problem of this electrificationin order to prevent electrification in a magnetic layerthe method of adding an additive is usually takenand the method of adding carbon black especially is the most effectiveand is adopted widely. Howeverif it was in the magnetic recording medium for the aforementioned high density recordingsince the fall of the output which lowers a magnetic body packing degree was causedaddition of carbon black has a limit in the additionand sufficient dealing with the prevention from electrification of it was not

completed.
[0011]Especially the aforementioned ferromagnetic hexagonal ferrite powderIt compares with Co-Fe_2O_3 ferromagnetic powderferromagnetic metal powderetc.Since saturation magnetization quantity is low and a high output is hard to be obtainedfor providing a high-output magnetic recording mediummust raise pack densitybut in hard [slight / it is particles and hard shape is a hexagonal form]. It is fundamentally difficult for dispersibility to be inferior as compared with conventional ferromagnetic powderand to secure antistatic property and a high reproducing output.
[0012]In order to satisfy prevention of electrification of the abovea high increase in powerand improvement in endurancethe proposal is indicated variously. JP55-55431AJP55-55432Aand JP55-55433A. Although an interlayer is provided between magnetic layerssuch as JP55-55434AJP60-164926AJP55-55436AJP62-38523Aand JP62-159337Aand a base materialCarbon black and bonding agent resin tend to be applied to an interlayerand it is going to form a magnetic layer on it after that.
[0013]Howeveralthough this method was effective in improvement dried cuttlefish in running durabilityit was a magnetic recording medium of high density recordingand moreoverthe outstanding magnetic parametric performancei.e.a high reproducing outputand an overwrite characteristic were not able to be satisfiedsecuring sufficient running durability. And the effective method of coping with this problem is not yet proposed.
[0014]
[Problem(s) to be Solved by the Invention]An object of this invention is to provide the running durability outstanding super-high-density magnetic recording medium that are made in view of the problem of said conventional technologyand magnetic parametric performancessuch as a high reproducing output and an overwrite characteristicare goodand are not charged easily.
[0015]
[Means for Solving the Problem]In a magnetic recording medium with which it comes to form a magnetic layer whichas for this inventionmakes a subject a non-magnetic layerferromagnetic powderand bonding agent resin which carry out nonmagnetic powder and bonding agent resin with a subject on a nonmagnetic substrate in this orderIt can consist of two-layer at leastand mean particle diameter of said lower layer ferromagnetic powder is larger than mean particle diameter of ferromagnetic powder of said upper layerand said magnetic layer is a magnetic recording mediumwherein overall thickness of this magnetic layer is [of the upper layer and a lower layer] 0.8 micrometer or lessandtherebycan solve an aforementioned problem.
[0016]It is the magnetic recording medium which provided a non-magnetic layer and a magnetic layer on a nonmagnetic substrate at this orderand a magnetic layer is the multiple layer structure more than two-layerand this invention limited overall thickness of that magnetic layer to 0.8 micrometer or less. A magnetic layer was made [2nd] into multiple layer structureand mean particle diameter of lower layer ferromagnetic powder was made larger than that upper.
[0017]By the waygenerallyalthough an output of ferromagnetic powder with small mean particle diameter is low in a recording wavelength region of long wavelengthas for particles with a high output in a short wavelength regionand large mean particle diameterthe reverse thing is known. It is known that a record signal of long wavelength is valid till a deep place of a magnetic layer. It constitutes from this invention so that the above-mentioned character whose frequency characteristic of ferromagnetic powder by difference in mean particle diameter is characteristic respectively in a lower layer and the upper layer of a magnetic layer may be demonstrated.
[0018]By making thickness of a magnetic layer thinthis invention reduced self-demagnetization lossand improved a reproducing outputand Namelythe overwrite characteristicFor examplethe characteristic that the latter signal is recorded effectively without being influenced by recording magnetization of the former signal even if it records a signal of short wavelength afterwards on a signal of long wavelength recorded before is improved. Although a coating method for this invention to form a magnetic layer and a non-magnetic layer does not have restriction in particularpreferablyIf a non-magnetic layer and a magnetic layer are formed with a wet-on-wet coating methoda thick taste is uniformand since the surface can form a magnetic layer whose adhesion with a non-magnetic layer of a smooth deer is also gooda magnetic recording medium with a high reproducing output which was excellent in endurance is provided.
[0019]It can apply and a magnetic layer can also be formedafter drying a non-magnetic layer. Howeverit is hard to secure the surface nature of a magnetic layerand when extremean application defect of a pinhole etc. may be produced in a magnetic layer. In this inventiona magnetic layer consists of two-layer at leastthe upper layer is a layer which consists of one or more layers which have at least a magnetic layer surface which separates most and is formed from a non-magnetic layerand a lower layer is a layer which consists of one or more layers formed between a non-magnetic layer and this upper layer at least.
[0020]Preferably0.01-0.7 micrometera range of the upper thickness is 0.05-0.4 micrometer still more preferablyand lower layer thickness0.1-0.7 micrometerit is the range of 0.4-0.75 micrometer preferablyand as for magnetic layer overall thicknessin order to secure 0.8 micrometer or lessespecially a recording wavelength of 1.4 micrometers or lessit is preferred that it is 0.5 micrometer or less. Overall thickness of a magnetic layer is written with a thin layer of 0.8 micrometer or lessand an overwrite characteristic peculiar to a digital recording can improve substantially. Since a recording wavelength can be shortened with 1.4 micrometers or lesslinear recording density can be improved and an effect by magnetic layer thickness reduction is demonstrated.
[0021]In this inventionif a relation of mean particle diameter of ferromagnetic powder which is with the upper layer

and a lower layer and is contained in each is satisfiedmultiple layer structure may be respectively sufficient as the upper layer and a lower layerand mean particle diameter of ferromagnetic powder in the upper layer and a lower layer may be different between each class in the upper layer and a lower layer in that case. For examplea magnetic layer may be constituted so that mean particle diameter of ferromagnetic powder may be gradually increased in a lower layer from the upper layer.

[0022]In this inventionmean particle diameter of ferromagnetic powder contained in the upper layer and a lower layer of a magnetic layer is the value which meant an average of an overall diameter of particles and was measured with an electron microscope. As for this upper content ferromagnetic powderexistence distribution in each class is controlled by this invention so that the mean particle diameter becomes small compared with this lower layer content ferromagnetic powder. In a magnetic recording medium of this inventionalthough iron oxide system ferromagnetic powderferromagnetic metal powderor ferromagnetic hexagonal ferrite powder can be used for ferromagnetic powder contained in a magnetic layerferromagnetic metal powder or ferromagnetic hexagonal ferrite powder is especially preferred.

[0023]In particularit is saturation magnetization ($sigma_S$) to a lower layer. High power can be obtained if large ferromagnetic metal powder is used. If ferromagnetic metal powder whose mean particle diameter is smaller than ferromagnetic metal powder used in a lower layer on itor ferromagnetic hexagonal ferrite powder is usedit will excel in the record/reproducing characteristics in short wavelength furtherand high power in a broad recording wavelength zone and high S/N will be obtained.

[0024]When ferromagnetic powder uses it for a lower layer of a magnetic layer with ferromagnetic metal powderthe mean particle diameter (long axis length)0.1-0.4 micrometerare the range of 0.12-0.35 micrometer preferablyand an acicular ratio (long axis length / minor-axis length)5-20and specific surface area are the range of 6-15 preferablyand according to a BET adsorption method -- 30-80m^2/g and crystallite size which is 35-70m^2/g preferably and is called for from an X-ray diffraction method -- 100-450 -- it is 150-350A preferably.

[0025]When ferromagnetic powder uses it for the upper layer of a magnetic layer with ferromagnetic metal powderthe mean particle diameter (long axis length)0.1-0.35 micrometerare the range of 0.12-0.30 micrometer preferablyand an acicular ratio (long axis length / minor-axis length)30-80m^2/g and 100-400 A of crystallite sizes of 5-20and specific surface area are the range of 6-15 preferablyand according to a BET adsorption method which are 40-70m^2/g preferably and are called for from an X-ray diffraction method are 120-330A preferably. Herecrystallite size is called for from breadth of half-the-price width of a diffraction line of a field (110) and a field (220).

[0026]When ferromagnetic powder uses it for a lower layer of a magnetic layer with ferromagnetic hexagonal ferrite powderthe mean particle diameter (plate diameter)0.01-0.2 micrometer is the range of 0.03-0.1 micrometer preferably -- a tabular ratio (a plate diameter/board thickness) -- 2-20and specific surface area are the range of 3-15 preferablyand according to a BET adsorption method -- 20-100m^2/g -- it is 25-80m^2/g preferably.

[0027]When ferromagnetic powder uses it for the upper layer of a magnetic layer with ferromagnetic hexagonal ferrite powderthe mean particle diameter (plate diameter)0.01-0.15 micrometer is the range of 0.02-0.12 micrometer preferably -- a tabular ratio (a plate diameter/board thickness) -- 2-20 -- it is the range of 3-15 preferably -- specific surface area -- 20-100m^2/g -- it is 25-80m^2/g preferably.

[0028]Since dispersibility worsens and surface nature is inferior when mean particle diameter of the upper ferromagnetic powder is smaller than the aboveit is not desirable. Since surface nature will worsen and an output will decline if mean particle diameter of lower layer ferromagnetic powder is larger than the aboveit is not desirable. When mean particle diameter of ferromagnetic powder used for the upper layer is larger than mean particle diameter of lower layer ferromagnetic powderit stops being suitable for high density recording.

[0029]In particulara kind of each ferromagnetic powder used for the upper layer and a lower layer is arbitrary without restrictionand if size relation of the above-mentioned mean particle diameter is satisfiedeven when it is of the same kindeven if it uses two or more sorts on different species or each classit will not be cared about. A magnetic recording medium of performance according to the purpose can be manufactured by selecting a kind of ferromagnetic powder in the upper layer and a lower layer. For exampleferromagnetic hexagonal ferrite powder etc. are listed in ** upper layer and a lower layer at ferromagnetic metal powder and ** upper layerand they are listed in ferromagnetic hexagonal ferrite powder and a lower layer at ferromagnetic metal powder** upper layerand a lower layer. When a magnetic layer is three or more layersin a layer system in the upper layer or a lower layertwo or more ferromagnetic powder of a different kind may be used by mixing or a layer unit.

[0030]Powder which contains Fe at least preferably is mentionedandspecificallysaid ferromagnetic metal powder has a metal simple substance or an alloy which made a subject FeFe-CoFe-nickelor Fe-nickel-Co. In order to high-recording-density-ize a magnetic recording medium of this inventionwhile it is required to control distribution about mean particle diameter within a magnetic layer of the above ferromagnetic metal powderas magnetic propertiesSaturation magnetization ($sigma_S$) At least 110 or more emu/g is 120 or more emu/g desirably. As coercive forcemore than 800Oe (oersted) is 900 or more Oe desirably. When ferromagnetic metal powder is used for the upper layer and a lower layerthe upper coercive force is equivalent to lower layer coercive forceor it is preferred that it is more than it.

[0031]In order to improve the characteristicnonmetalssuch as BCaluminumSiand Pmay be added during a presentation. Usuallya layer of an oxide is formed in order to stabilize a particle surface of said metal powder chemically. How to dry after being immersed in a publicly known gradual oxidation treatmenti.e.an organic solventas a formation method of an oxideA method of dryingafter sending in oxygen containing gas after being immersed in an organic solventand

forming an oxide film in the surfacea method of adjusting a partial pressure of oxygen gas and inactive gas not using an organic solventand forming an oxide film in the surfaceetc. are mentionedand what gave any can be used.
[0032]As said ferromagnetic hexagonal ferrite powderIt is ferromagnetic powder which has an easy axis in the direction vertical to the monotonous side by plate-likeThere are a barium ferritea strontium ferritea lead ferritecalcium ferritesor those cobalt substitution productsand a cobalt substitution product of a barium ferrite and a cobalt substitution product of a strontium ferrite are preferred also in especially inside. In order to improve the characteristic if neededelementssuch as InZngermaniumNband Vmay be added. In order to high-recording-density-ize a magnetic recording medium of this inventionwhile it is required to control grain size distribution in a magnetic layer of said hexagonal ferrite powder in the aforementioned relation in the upper layer and a lower layeras magnetic propertiesSaturation magnetization ($sigma_s$) At least 50 or more emu/g is 53 or more emu/g desirably. Especially as coercive forceit is desirable that they are 600 or more Oe 500 or more Oe. There is the feature that the hexagonal ferrite powder can expect high power rather than other magnetic particles if 1.5 micrometers or less of recording wavelengths of a high frequency band serve as short wavelength record of 1.0 micrometer or less preferably in proportion to a magnetic particle of others [output]although it is low in long wavelength record.
[0033]In a magnetic recording medium of this inventionif it is in a magnetic recording medium of disk form like a magnetic recording diskan output of a circumferencial direction is uniformno change is wanted for there to beit is needed that an in-plane orientation degree ratio is high as much as possible for that purposeand an amount-of-preferred-orientation ratio of ferromagnetic powder is 0.85 or more preferably. A random orientation method or JP63-148417A which uses a permanent magnet like JP3-41895B in a place which a magnetic layer has in a state where it does not dry in order to make an amount-of-preferred-orientation ratio or more into 0.85A method of impressing an alternating current magnetic field can be used like each gazettesuch as JP1-300427A and JP1-300428A.
[0034]In this inventionif hexagonal ferrite powder is used0.9 or more high amount-of-preferred-orientation ratios are realizable. Herean amount-of-preferred-orientation ratio is the value which **(ed) the minimum square-shaped ratio of a circumferencial direction by a maximum angle form ratio. The magnetic properties of ferromagnetic powdersuch as saturation magnetization quantity and coercive forceand an amount-of-preferred-orientation ratio were measured by maximum impression magnetic field 5kOe using an oscillating sample type flux meter (made by Toei Industry). Measurement of specific surface area is based on a BET adsorption method which used KANTA soap (made in [KANTA chromium company] the U.S.). They are 250 ** and the value measured by a nitrogen atmosphere for 30 minutes with an after-drying BET one point method for BET method (partial pressure 0.30).
[0035]As for water content of these ferromagnetic powderit is preferred to consider it as 0.01 to 2 % of the weight. As for water contentit is preferred to optimize according to a kind of bonding agent resin. It is preferred to optimize with combination with bonding agent resin which pH of ferromagnetic powder also uses. Although the range is 4-12it is 5-10 preferably. Ferromagnetic powder may perform a surface treatment with aluminumSiPor these oxides if needed. When the quantity is 0.1 to 10% and a surface treatment is performed to ferromagnetic powderadsorption of lubricantsuch as fatty acidbecomes below 100mg[/m] ^{2}and is preferred. Although inorganic ionsuch as Na of fusibilityCaFenickeland Srmay be included in ferromagnetic powderthe characteristic will not be affected especially if it is 500 ppm or less.
[0036]If iron oxide ferromagnetic powder may be used by request and it expresses with specific surface area by a BET adsorption method as ferromagnetic powderit will be 25-80m^2/g and will be 35-60m^2/g preferably. A noise becomes highand it is hard to obtain surface nature above 80-m^2/gand is not desirable at below 25-m^2/g. Crystallite size measured by an X-ray diffraction method is 450-100 Aand is 350-100 A preferably. 50 or more emu/g of $sigma_s$ are 70 or more emu/g preferably.
[0037]You may process beforehand before distribution with a dispersing agentlubricanta surface-active agenta spray for preventing static electricityetc. which are stated to these ferromagnetic powder later. Specificallyit is indicated to JP44-14090B etc. A non-magnetic layer of a magnetic recording medium of this invention makes a subject nonmagnetic powder and bonding agent resin. As for nonmagnetic powderit is preferred that include inorganic powder and organic powder and inorganic powder is included at leastand it is preferred that carbon black is included as organic powder.
[0038]3 to 20% of the weight of the nonmagnetic powder whole quantity of content of carbon black in a non-magnetic layer contained in a non-magnetic layer is preferred. Although a surface specific resistance value cannot fully be reduced and reduction of surface specific resistance value sufficient at 20 % of the weight or more can be performed at 3 or less % of the weightthe surface nature of a magnetic layer smooth enough cannot be obtained. 5-1500-m^2/g of specific surface area is the range of 700-1400-m^2/g desirable still more preferably.
[0039]Therebyan addition of carbon black in a magnetic layer can be reduced. Since this carbon black forms structureit can obtain low surface electric resistance. For this reasona surface characteristic-electric-resistance value of a magnetic layer can also be held down lowand generating of a dropout in running durability can be reduced. Carbon black whose mean particle diameter is less than 40mmicro and whose DBP oil absorption is 300 mL(s) (milliliter)/100g as desirable carbon black is mentioned especially. Therebysince mean particle diameter is less than 40mmicrothe smooth surface nature of a magnetic layer lower layer / upper layer is obtainedspacing loss with record/playback head can be lessenedand a high reproducing output is obtained. DBP oil absorption can obtain surface electric resistance low as a result that it is easy to form structureand can reduce especially generating of a dropout in running durabilityand 300 mL(s)/not less than 100g of carbon black has it. [still more preferred]

[0040]DBP oil absorption of carbon black adds dibutyl phthalate to carbon black powder little by littleA state of carbon black was observed making it scour each othera point of making one lump from a state distributed scatteringly was found outand an addition (mL) of dibutyl phthalate at that time was made into DBP oil absorption. It is effective in order to give conductivity to a magnetic recording medium and to prevent electrification of a magnetic recording mediumand carbon black is used also as a raw material for adjusting physical intensity of a magnetic layer and a non-magnetic layer. Carbon black also has a function to adjust the viscoelastic property of coating liquid for non-magnetic layers. Carbon black is a very useful raw material which has various functionssuch as adjustment of a coefficient of frictionand light blocking effect grant. Thereforeit is preferred to include carbon black also in a magnetic layer with the same main point as the above.
[0041]This invention can use a thing of all processes as carbon black which can be used for a non-magnetic layer. For examplefurnace blackthermal blackacetylene blackchannel blacklamp blacketc. can be used. As a concrete example#3950B by a Mitsubishi Kasei industrial companyKetchen black EC by lion AkzoKetchen black ECDJ-500Ketchen black ECDJ-600etc. are mentioned.
[0042]It does not matter even if it uses what graphite-ized surface [a part of] even if the surface treatment was carried outor it used carbon black by resin with a dispersing agent etc.having graft-ized it. Before adding carbon black to a nonmagnetic coating materiala binding material may distribute beforehand. these carbon black -- independence -- or it can be combined and used.
[0043]The carbon black which can be used by this invention can refer to (a volume "carbon black manual" and for carbon black associations). a magnetic recording medium of this invention -- a wet-on-wet coating method -- that isOn a nonmagnetic substrateapply coating liquid for non-magnetic layersand a coating layer for non-magnetic layers is formedIt is preferred to manufacture by applying coating liquid for lower layers of a magnetic layer on said coating layer for non-magnetic layerswhile this coating layer for non-magnetic layers is in a damp or wet conditionand applying coating liquid for the upper layers of a magnetic layer on said coating layer for lower layerswhile this coating layer for lower layers is in a damp or wet condition further.
[0044]even if this wet-on-wet coating method applies each coating liquid simultaneously substantially in time -- things -- even if it applies with a time intervaleach coating liquid should just be a damp or wet condition. Even if a magnetic layer comprises three or more layersa fundamental concept of the above-mentioned spreading is the same. If an ultra-thin magnetic layer with uniform thickness is obtained.and a wet-on-wet coating method has thin thickness of a magnetic layerthe adhesion of the upper layer of the lower layer/magnetic layer of the non-magnetic layer/magnetic layer which poses a problem will be improved. Magnetic layer overall thickness can prevent peeling of a magnetic layer of 0.8 micrometer or lessand this coating method can obtain an outstanding magnetic recording medium of running durability which a dropout does not produce easily. Probably because the magnetic layer is very thina method which applies a magnetic layer on it after applying coating liquid of a non-magnetic layerdrying and forming a non-magnetic layer is not enough as the adhesion of a non-magnetic layer and a magnetic layerand each class cannot become one structure easily by it as a layer formed on a nonmagnetic substrate.
[0045]There is viscoelastic property (thixotropy) of coating liquid as what should be minded by a wet-on-wet method. That iswhen a difference of the viscoelastic property of each coating liquid of a magnetic layer and a non-magnetic layer was large and it applieslike this inventionwhen thickness of a magnetic layer is very thinit is easy for mixture **** of liquid to happen by an interface of each classand to cause a problem of the surface nature of a magnetic layer falling.
[0046]In order to bring the viscoelastic property of coating liquid close as much as possibleit is effective to make the same first a particulate material of each coating liquid of a magnetic layer and a non-magnetic layerbut. In the case of this inventionsince it is not madein order to double with structural viscosity which structure structure where ferromagnetic powder is formed of magnetism in coating liquid of a magnetic layer brings aboutit is desirable to use particles which are easy to form structural viscosity like carbon black as a nonmagnetic particle of non-magnetic layer coating liquid. Thereforein this inventionalthough it is effective that oil absorption uses large carbon black with a small grain sizeit is also effective to use nonmagnetic inorganic powder with simultaneously small grain size other than carbon black. For examplein particlessuch as 1 micrometer or less of titanium oxideand oxidation aluminumit is easy to become coating liquid with the structural viscosity of particles by moderate condensation.
[0047]As for nonmagnetic inorganic powder which can be used for a non-magnetic layer of this inventionmetala metallic oxidemetallic carbonatemetal sulfatemetal nitridemetallic carbidemetallic sulfideetc. are mentionedfor example. Specifically TiO_2 (rutileanatase)TiO_XCerium oxidetin oxidetungstic oxide$ZnOZrO_2SiO_2$ and Cr_2O_3alpha alumina as for which not less than 90% of a pregelatinization rate getsBeta-aluminagamma aluminaalpha-oxidation irongoethitecorundumsilicon nitrideThey are usedtitanium carbidemagnesium oxideboron nitridea molybdenum disulfidecopper oxide$MgCO_3CaCO_3BaCO_3SrCO_3BaSO_4CaSO_4$silicon carbideetc. being independentor combining. Shape of these inorganic powder and size are arbitrary at a needlea globular shapedie shapeetc.these can combine inorganic powder different if neededor independent nonmagnetic powder can also choose particle size distribution etc. It is chosen from 0.01-2 micrometers as a grain size. As nonmagnetic powderthe following are preferred.
[0048]pH of 1-30-m^2/g of 2-11and specific surface area is [0.3-2g /and water content cc] preferred [tap density] 0.1 to 5%. 5-100ml/100 g of oil absorption [10-80ml/100 g of] using DBP is 20-60 ml/100 g still more preferably preferably. The above-mentioned nonmagnetic powder does not necessarily need to be pure 100%may process the surface according to the purpose with each compoundsuch as other compoundsfor examplealuminumSiTiZrSnSband

Znand may form those oxides in the surface. In that caseif purity is not less than 70%it will not become reducing an effect. As for ignition lossit is preferred that it is 20% or less.

[0049]As a concrete example of nonmagnetic powder used for this inventionAKP-20 by Sumitomo Chemical Co.Ltd.AKP-30AKP-50the Nippon Chemical Industrial Co.Ltd. make G5G7S-1TF-100 by Toda Kogyo Corp.TF-120TF-140etc. are mentioned. Although acrylic styrene resin powderbenzoguanamine resin powdermelamine system resin powderand phthalocyanine pigment are mentionednonmagnetic organic powder used for this inventionPolyolefin-system-resin powderpolyester system resin powderpolyamide system resin powderpolyimide system resin powderand polyfluoroethylene resin powder are used. What is indicated in each gazette of JP62-18564A and 60-255827 can be used for the process.

[0050]As a binding material used for a magnetic layer of this inventionand a non-magnetic layerpublicly known thermoplastic system resinheat-curing system resinreaction type resinand these mixtures are used conventionally. As thermoplastic system resinglass transition temperature is -100-150 **and number average molecular weights are 1000-200000and a thing 10000-100000and whose degree of polymerization are about 50 to about 1000 preferably.

[0051]As such an exampleVCM/PVCvinyl acetatevinyl alcoholMaleic acidacrylic acidacrylic estera vinylidene chlorideacrylonitrileThere are a polymer or a copolymerpolyurethane resinand various rubber system resin which contain methacrylic acidmethacrylic acid esterstyrenebutadieneethylenevinylbutyrala vinyl acetalvinyl etheretc. as a constitutional unit.

[0052]As thermosetting resin or reaction type resinphenol resinan epoxy resinPolyurethane hardening resinurea resinmelamine resinalkyd resinA mixture of acrylic reaction resinformaldehyde resinssilicone resinepoxy-polyamide resinpolyester resinand an isocyanate prepolymera mixture of polyester polyol and polyisocyanatea mixture of polyurethane and polyisocyanateetc. are raised.

[0053]These resin is indicated in detail in a "plastic handbook" of the Asakura Publishing issue. It is also possible to use publicly known electron beam hardening resin for a lower layer or the upper layer. These example and its manufacturing method are indicated in detail to JP62-256219A. Although it can be used the above resin being able to be independent or combiningAs a desirable thingvinyl chloride resinVCM/PVC vinyl acetate resinVCM/PVC vinyl acetate vinyl alcohol resinCombination of at least one sort and polyurethane resin which are chosen from a group of a VCM/PVC vinyl acetate maleic anhydride copolymeror a thing which combined polyisocyanate with these is raised.

[0054]The structure of polyurethane resin can use publicly known thingssuch as polyester polyurethanepolyether polyurethanepolyether polyester polyurethanepolycarbonate polyurethanepolyester polycarbonate polyurethaneand polycaprolactone polyurethane. In order to obtain more outstanding dispersibility and endurance about all the binding materials shown hereif needed. $COOMSO_3MOSO_3MP{=}O(OM)_2O\text{-}P{=}O(OM)_2$ -- (-- per above -- M -- hydrogen atom or alkaline metal). As for $OHNR_2$and N^+R_3 and (Rit is preferred to use what introduced at least one or more polar groups chosen from hydrocarbon-group)an epoxy groupSHCNetc. by copolymerization or an addition reaction. Quantity of such a polar group is 10^{-1} - 10^{-8} mol /gand is 10^{-2} - 10^{-6} mol /g preferably.

[0055]As a concrete example of these binding materials used for this invention:VAGH by a union carbide companyVYHHVMCHVAGFVAGDVROHVYESVYNCVMCCXYHLXYSGPKHHPKHJPKHCPKFENissin Chemical Industry Co.Ltd. make : MPR-TAMPR-TA5MPR- TALMPR-TSNMPR-TMFMPR-TSMPR-TMMPR-TAOElectrochemistry company make : 1000WDX80DX81DX82DX83100FDNippon Zeon Co.Ltd. make : MR105MR110MR100400X110AJapanese polyurethane company make :. Made in [Toyobo Co.Ltd.] NIPPORAN N2301N2302N2304Dainippon Ink:bread DEKKUSU T-5105T-R3080T-5201bar knock D-400and D-210-80 and Chris Bon 6109 and 7209 : Byran UR8200UR8300UR8600UR5500UR4300RV530RV280great Nissei-ized company make : DAIFUE lamin 4020502051005309090209022and 7020 and :MX5004 by Mitsubishi Kasei Corp.Mitsuhiro -- transformation -- shrine: -- Sun Plane SP-150the Asahi Chemical Co.Ltd. make:saran F310F210etc. are raised.

[0056]a binding material in which a binding material used for a magnetic layer of this invention is used for a non-magnetic layer as opposed to ferromagnetic powder receives nonmagnetic powder -- 5 to 50% of the weight of a range -- it is preferably used in 10 to 30% of the weight of the range. When using VCM/PVC system resinit is preferred to combine in the range in 2 to 50 % of the weightwhen using polyurethane resin five to 100% of the weightand for polyisocyanate to combine these in 2 to 100% of the weight of the rangeand to use.

[0057]In this inventionrupture stress is 0.05 - 10 kg/cm^{2}and a breakdown point's glass transition temperature is [-50-100 ** and elongation after fracture / 0.05 - 10 kg/cm^2] preferred 100 to 2000%when using polyurethane resin. A magnetic recording medium of this invention consists of the upper layer of a non-magnetic layer and a magnetic layerand three lower layer layers or more fundamentally. A non-magnetic layerthe upper layerand a lower layer may be respectively formed in two or more layers. Thereforeof courseit is possible to change VCM/PVC system resinpolyurethane resin and polyisocyanate which are occupied in the amount of binding materials and a binding material or quantity of the other resina molecular weight of each resin which forms a magnetic layerthe amount of polar groupsor the physical property of resin described previously on each class if needed.

[0058]As polyisocyanate used for this inventionTolylene diisocyanate4-4'-diphenylmethane diisocyanateHexamethylene di-isocyanatexylylene diisocyanatenaphthylene-15-diisocyanateIsocyanatessuch as an o-toluidine isocyanateisophorone diisocyanateand triphenylmethane triisocyanate. Polyisocyanate etc. which were generated according to output with such isocyanates and polyalcohols and condensation of isocyanates can be used. As a trade name with which these isocyanates are marketedJapanese polyurethane company make : The coronate Lthe coronate HLthe coronate 2030the coronate 2031milli ONETO MRMilli ONETO MTLTakeda ChemicalLtd. make :

Takenate D-102 and Takenate D-110NTakenate D-200Takenate D-202the Sumitomo Beyer company make: There are Desmodur LDesmodur ILDesmodur NDesmodur HLetc.and these can be used for each class in two or combination beyond it using an independent or hardening reactant difference.

[0059]The carbon black used for a magnetic layer of this invention can use a furnace for rubbersblack for thermal ** colors for rubbersacetylene blacketc. specific surface area -- 10-1500 ml/100 gand particle diameter are 5mmicro-300mmicroand pH of tap density is [5-500m^2/g and DBP oil absorption / 2-10and water content] preferred in 0.1-1g/cc 0.1 to 10%. As a concrete example of carbon black used for this invention. ** Cabot Corp. make : BLACKPEARLS 2000130010009008O0and 700VULCAN XC-72:**80 by Asahi carbon company**60**55**50**35and Mitsubishi Kasei industrial company make:**3950B**2400B**2300**900**1000**30**40**10BKONRON beer carbon company make : CONDUCTEX SCRAVEN 1505040and 15a product made by lion OKUZO: Ketchen black ECKetchen black ECDJ-500Ketchen black ECDJ-600etc. are mentioned. It does not matter even if it uses what graphite-ized surface [a part of] even if the surface treatment was carried outor it used carbon black by resin with a dispersing agent etc.having graft-ized it. Before adding carbon black to a magnetic painta binding material may distribute beforehand. These carbon black can be used in independence or combination. When using carbon blackit is preferred to use in 0.1 to 30% of quantity to ferromagnetic powder. Carbon black has work of prevention from electrification of a magnetic layercoefficient-of-friction reductionlight blocking effect grantimprovement in film strengthetc.and these change with carbon black to be used. Thereforeof courseit is possible to use properly according to the purpose based on various characteristics which these carbon black used for this invention changed the kindquantityand combination in a lower layer and the upper layerand were shown in the pointssuch as grain sizeoil absorptionelectric conductivityand pH. The carbon black which can be used by a magnetic layer of this invention can refer to a "carbon black manual" (volume for carbon black associations).

[0060]As abrasive soap used for a magnetic layer or a non-magnetic layer of this inventionalpha-alumina of not less than 90% of a pregelatinization ratebeta-aluminasilicon carbidechrome oxidelt is used in that a with a Mohs hardness [of five or more] publicly known material is mainly independentor combinationsuch as cerium oxidealpha-iron oxidecorunduma synthetic diamondsilicon nitridesilicon carbidetitanium carbidetitanium oxidea silicon dioxideand boron nitride. A complex (what carried out the surface treatment of the abrasive soap with other abrasive soap) of these abrasive soap may be used. Although compounds or elements other than the main ingredients may be contained in these abrasive soapif the main ingredients are not less than 90%there will be instead of [no] in an effect.

[0061]A thing of 0.05-5-micrometer ******** is effectiveand mean particle diameter of these abrasive soap is 0.2-1.0 micrometer preferably. Abrasive soap in which grain size differs if needed is combinableor independent abrasive soap can also make particle size distribution largeand can also give same effect. pH of 1-30-m^2/g** of 2-11and specific surface area is [0.3-2g /and water content cc] preferred [tap density] 0.1 to 5%. A needlea globular shapedie shapeand ******** may be sufficient as shape of abrasive soap used for this invention.

[0062]These abrasive soap is added in the range of three to 20 weight section to each bonding-agent-resin 100 weight section of a non-magnetic layer and a magnetic layer. If less than three weight sectionssufficient endurance will not be obtainedand if too more than 20 weight sectionsa packing degree will decrease and sufficient output will not be obtained. In a non-magnetic layerquantity of nonmagnetic powder to containa kindand a magnetic layer setthe combination is changed according to the upper layer and quantity of lower layer ferromagnetic powderand a kindandof coursethese abrasive soap can be properly used according to the purpose. For examplewhen raising the endurance of a magnetic layer surfaceand the amount of abrasive soap of a non-magnetic layer raises the endurance of the magnetic layer end faceit can devise increasing the amount of abrasive soap of a magnetic layer etc. After carrying out the distributed processing of these abrasive soap with a binding material beforehandit may be added in a magnetic paint and a nonmagnetic coating material. Five pieces/more than 100micrometer^2 of abrasive soap which exists in a magnetic layer surface and the magnetic layer end face of a magnetic recording medium of this invention is preferred.

[0063]As a concrete example of abrasive soap used for this inventionSumitomo Chemical Co.Ltd. make:AKP-20AKP-30AKP-50HIT-50:G5 by Nippon Chemical Industrial Co.Ltd.G7S-1Toda Kogyo Corp. make:TF-100TF-140100ED140EDetc. are raised. As a dispersing agent (paints wetting agent) used for this inventionCaprylic acidcapric acidlauric acidmyristic acidpulmitic acidStearic acidbehenic acidoleic acidelaidic acidlinolic acidFatty acid of 12-18 carbon numbers of linolenic acidsteer roll acidetc. (it R_1-COOH(s) and) R_1 -- alkyl of 11-17 carbon numbersor alkenyl group; -- an alkaline metal () of the aforementioned fatty acid [Li and] alkaline-earth metals (Mg and Ca.)such as Na and K compound; containing fluorine of fatty acid ester of the metallic soap; above which consists of Ba) -- amide [of said fatty acid]; -- poly ARIKIREN oxide alkyl-phosphoric-acid ester; -- lecithin; -- trialkyl polyolefine oxy quarternary ammonium salt (1-5 carbon numbers alkyl) As for an olefin;such as ethylene and propyleneetc. are used. In additionit is usable besides these in with a carbon numbers of 12 or more higher alcoholsulfate esteretc. These dispersing agents are added in the range of 0.5 to 20 weight section to condensing agent resin 100 weight section.

[0064]As lubricanta dialkyl polysiloxane (alkyls are 1-5 carbon numbers)A dialkoxy polysiloxane (alkoxy groups are 1-4 carbon numbers)a monoalkyl monoalkoxy polysiloxane (1-5 carbon numbers alkyl) An alkoxy group Conductive impalpable powder; molybdenum disulfidesuch as silicone oil; graphitesuch as 1-4 carbon numbersa phenylpolysiloxaneand a phloroalkyl polysiloxane (alkyl groups are 1-5 carbon numbers)Inorganic powdersuch as a tungsten disulfide; Polyethylenepolypropyleneunsaturation aliphatic hydrocarbon (a compound which n-olefinic linkage combined with carbon of an end.) liquefied at plastic impalpable powder; alpha olefin polymer; ordinary

temperaturesuch as a polyethylene vinyl chloride copolymer and polytetrafluoroethylene Carbon number 20 [about]; the fatty acid ester and fluorocarbon etc. which comprise monobasic fatty acid of 12-20 carbon numbers and alcohol of monovalence of 3-12 carbon numbers can be used.

[0065]Fatty acid ester is especially the most preferred. As alcohol used as a raw material of fatty acid esterEthanolbutanolphenolbenzyl alcohol2-methylbutyl alcohol2-hexyldecyl alcoholpropylene glycol monobutyl etherEthylene glycol monobutyl etherdipropyleneglycol monobutyl etherPolyhydric alcoholsuch as monoalcoholsuch as diethylene-glycol monobutyl ether and sec-butyl alcoholethylene glycola diethylene glycolneopentyl glycolglycerinand a sorbitan derivativeis mentioned.

[0066]Similarly as fatty acidacetic acidpropionic acidoctanoic acid2-ethylhexanoic acidAliphatic carboxylic acid or these mixturessuch as lauric acidmyristic acidstearic acidpulmitic acidbehenic acidarachin acidoleic acidlinolic acidlinolenic acidelaidic acidand palmitoleic acidare mentioned. An example as fatty acid ester Butyl stearates-butyl stearateIsopropyl stearatebutyl oleateamyl stearate3-methylbutyl stearate2-ethylhexyl stearate2-hexyldecyl stearatebutyl palmitate2-ethylhexyl Millis TaitButyl stearateand a mixture of butyl palmitatebutoxyethyl stearate2-butoxy-1-propyl stearatea thing which acylated dipropyleneglycol monobutyl ether with stearic acidVarious ester compoundssuch as oleate etc. of what acylated diethylene-glycol dipalmitate and hexamethylenediol with myristic acidand was made into diesterand glycerincan be mentioned.

[0067]When using a magnetic recording medium under high humiditychoosing opposite-sex structuressuch as fatty acid of a raw material and branching/straight chain of alcoholcis- one/transformerand a branch location as a ****** sake is made in hydrolysis of often produced fatty acid ester. These lubricant is added in the range of 0.2 to 20 weight section to bonding-agent-resin 100 weight section.

[0068]As lubricantthe following compounds can also be used further. That isthey are a silicone oilgraphitemolybdenum disulfideboron nitridegraphite fluoridefluoride alcoholpolyolefinepolyglycolalkyl phosphoric estera tungsten disulfideetc. These lubricant used by this invention can use the kind and quantity properly if needed by magnetic layer and a non-magnetic layer. For exampleusing fatty acid in which the melting points differ on each classusing ester species from which the boiling point to the surface which oozes and controls **and polarity differan addition of lubricant to the surface which oozes and controls ** is increased by a non-magnetic layerand it is possible to raise a lubrication effect etc. and is not restricted only to an example shown here of course.

[0069]All the additive agents used by this invention or its partWhen you may addfor exampleit mixes with ferromagnetic powder before a mixing step at which process of magnetic paint manufactureit adds by a mixing step by ferromagnetic powdera binding materialand a solventit adds by a dispersing process and it adds after distributionit may add just before spreading. As an example of goods of these lubricant used by this inventionNippon Oil & Fats Co.Ltd. make :. NAA-102NAA-415NAA-312NAA-160NAA-180NAA-174NAA-175NAA-222NAA-34NAA-35NAA-171NAA-122NAA-142NAA-160NAA-173KCastor bean hardening fatty acidNAA-42NAA-44cation SAcation MAthe cation ABcation BBNAIMIN L-201NAIMIN L-202NAIMIN S-202Nonion E-208Nonion P-208Nonion S-207Nonion K-204Nonion NS-202Nonion NS-210Nonion HS-206Nonion L-2Nonion S-2Nonion S-4Nonion O-2Nonion LP gas-20RNonion PP-40RNonion SP-60RNonion OP-80RNonion OP-85RNonion LT-221Nonion ST-221Nonion OT-221MONOGURI MBNonion DS-60anone BFthe anone LGbutyl stearatebutyl laurateerucic acidKanto Kagaku make : Oleic acidThe Takemoto fats-and-oils company make :. FAL-205FAL-123New Japan Chemical Co.Ltd. make :. NG -- grass -- LOENUJORUBU IPMthe SANSO sizer E4030Shin-etsu chemicals company make:TA-3and KF-96 and KF-96LKF-96HKF410KF420KF965KF54KF50KF56KF-907KF-851and X-22-819. X-22-822KF-905KF-700KF-393KF-857KF-860KF-865X-22-980KF-101KF-102KF-103X-22-3710X-22-3715KF-910KF-3935Made in lion Armagh : Armide PArmide CAMO slip CPLion fats-and-oils company make : DEYUOMIN TDONisshin Oil MillsLtd. make:BA-41GMitsuhiro -- transformation -- shrine: -- pro fan 2012E and new pole PE61 and Io -- net MS-400 and Io -- net MO-200 and Io -- net DL-200 and Io -- net DS-300 and Io -- net DS-1000 and Io -- net DO-200 etc. is raised.

[0070]An organic solvent used by this invention by arbitrary ratios Acetonemethyl ethyl ketoneMethyl isobutyl ketonediisobutyl ketonecyclohexanoneKetonesuch as isophorone and a tetrahydrofuranmethanolethanolPropanolbutanolisobutyl alcoholisopropyl alcoholAlcoholssuch as methyl cyclohexanolmethyl acetatebutyl acetateEster speciessuch as isobutyl acetateisopropyl acetateethyl lactateand acetic acid glycolGlycol wood etherglycol monoethyl etherdioxaneWhich glycol ether systembenzenetoluenexylenecresolThingssuch as chlorinated hydrocarbonsuch as aromatic hydrocarbonsuch as chlorobenzenemethylene chlorideethylene chloridea carbon tetrachloridechloroformethylene chlorohydrineand dichlorobenzeneN.N-dimethylformamideand hexanecan be used. These organic solvents are not necessarily pure 100%and non-puritysuch as an isomeran unreacted materiala side reaction thinga decomposition productan oxideand moisturemay be contained in addition to the main ingredients. 30 or less % of the weight is desirable still more preferredand such non-purity is 10 or less % of the weight.

[0071]As long as an organic solvent used by this invention is necessarythe kind and quantity may be changed on each class of a non-magnetic layer and a magnetic layer. a lower layer or a non-magnetic layer of a magnetic layer which uses a volatile high solvent for the upper layer of a magnetic layerand raises surface nature -- a solvent with high surface tension (cyclohexanone.) Although using a high solvent of a soluble parameter for this lower layer or a non-magnetic layer which raises the stability of spreading using dioxane etc.and raising a packing degree etc. is mentioned as the exampleit is undoubted that it is not what was restricted to these examples.

[0072]A nonmagnetic substrate used for this invention Polyethylene terephthalatePublicly known filmssuch as

polyestersuch as polyethylenenaphthalatepolyolefinescellulose triacetatepolycarbonatepolyamidepolyimidepolyamidoimidepolysulfonepolyether sulphonearamidand aromatic polyamidecan be used. As for a nonmagnetic substrategenerally1-100 micrometers of 25-85-micrometer-thick things are used preferably. Corona discharge treatmentplasma treatmenteasily-adhesive processingheat treatmentdust removing treatmentetc. may be beforehand performed to these base materials.

[0073]In order to attain the purpose of this inventionit is a center line average-surface-roughness (Ra) (cutoff value as a nonmagnetic substrate. It is desirable for 0.25mm) to use 0.03 micrometer or less of things [0.02 micrometer or less of] of 0.01 micrometer or less still more preferably preferably. As for these nonmagnetic substratesit is preferred that not only center line average surface roughness is smallbut there is no big and rough projection of 1 micrometers or more. Surface granularity shape is freely controlled with a size and quantity of a filler which are added by base material if needed. As these fillersorganic impalpable powdersuch as acrylic besides oxidessuch as CaSiand Tior carbonateis mentioned as an example.

[0074]Preferably F-5 value of a web running direction (longitudinal direction) of a nonmagnetic substrate used for this invention 5-50kg[/mm] ^{2}Although it is generalF-5 value of a web width direction is 3-30kg[/mm] ^{2} preferablyand when it is necessary to make especially crosswise intensity highthe limitation does not have F-5 value of a web longitudinal direction higher than F-5 value of a web width direction.

[0075]An under coat which consists of polyethylene resin for improvement in adhesionetc. may be provided between a nonmagnetic substrate and a non-magnetic layer. 0.01-2 micrometers of this thickness is 0.05-0.5 micrometer preferably. A back coat layer may be provided in an opposite hand the magnetic layer side of a nonmagnetic substrate. 0.1-2 micrometers of this thickness is 0.3-1.0 micrometer preferably. These under coats and the back coat layer can use a publicly known thing. In the case of a disc-like magnetic recording mediuma magnetic layer can be provided in both sides or one side.

[0076]Preferably3% or lessit is still more desirablea heat shrinkage rate for 1.5% or less and 80 ** 30 minutes is preferredand a heat shrinkage rate for 100 ** 30 minutes of a web running direction of a nonmagnetic substrate and the cross direction is 0.5% or less still more preferably 1% or less. Breaking strength is 5-100kg[/mm] ^{2}and 100-2000kg[/mm] ^{2} of an elastic modulus is [both directions] preferred.

[0077]A process of manufacturing a magnetic paint of a magnetic recording medium of this invention consists of a mixing stepa dispersing processand a mixing process established if needed before and after these processes at least. Each process may divide into two or more stepsrespectively. All the raw materialssuch as ferromagnetic powdera binding materialcarbon blackabrasive soapa spray for preventing static electricitylubricanta solventetc. which are used for this inventionmay be added in the middle of the beginning of which process. Each raw material may be divided at two or more processesand you may add. For examplepolyurethane may be divided and supplied by a mixing process for viscosity control after a mixing stepa dispersing processand distribution.

[0078]Various kinds of kneading machines are used in kneading dispersion of a magnetic paint. For example2 roll mills3 roll millsa ball milla pebble millA TRON milla Sand grinderZEGUBARI (Szegvari)attritora high-speed impeller dispersion machinea high-speed stone milla high-speed shock millDISUPAa kneadera high speed mixera homogenizeran ultrasonic dispersion machineetc. can be used.

[0079]In order to attain the purpose of this inventionof coursethe conventional publicly known production technology can be used as some processesbut in a mixing stephigh Br of a magnetic recording medium of this invention can be obtained by using a thing with strong kneading powersuch as a continuation kneader and a pressurized kneader. When using a continuation kneader or a pressurized kneaderkneading processing is carried out in 15-500 copies to all or its part (however30% of the weight or more of all the binding materials are preferred)and 100 copies of ferromagnetic powder of ferromagnetic powder and a binding material. Details of these kneading processings are indicated to JP1-106338A and JP64-79274A. When preparing non-magnetic layer liquidit is desirable to use high-density dispersion mediaand zirconia beads and a metal bead are preferred.

[0080]In this inventionit can produce more efficiently by using a simultaneous multistory coating method as shown in JP62-212933A. The following composition is mentioned as an example of a device which applies a magnetic recording medium of multistory composition like this inventionand a method.

1. With photogravure spreadingroll coatingbraid spreadingan extrusion coateretc. which are generally used by spreading of a magnetic paint. A way apply the 1st layer first and the 1st layer applies the 2nd layer with a base material pressurization mold extrusion coater currently indicated by each gazette of JP1-46186BJP60-238179Aand JP2-265672A in inside of a wet state.
2. How to apply the 1st layer and the 2nd layer almost simultaneous by one spreading head which builds in two coating liquid dipping slits which are indicated by each gazette of JP63-88080AJP2-17921Aand JP2-265672A.
3. How to apply the 1st layer and the 2nd layer almost simultaneous with extrusion coater with the back up roll currently indicated by JP2-174965A.

[0081]In order to prevent a fall of the magnetic parametric performance of a magnetic recording medium by condensation of ferromagnetic powderetc.it is desirable to give shearing to coating liquid inside a spreading head by a method which is indicated by JP62-95174A and JP1-236968A. An existing plastic roll of heat resistancesuch as epoxypolyimidepolyamideand polyimidoamideis used as a calendar treating roll. It can also process by a metallic roll comrade. Not less than 70 ** of treatment temperature is not less than 80 ** still more preferably preferably. Linear pressure power is 300 or more kg/cm still more preferably 200 kg/cm preferably.

[0082]Surface specific resistance of a magnetic layer side of a magnetic recording medium of this invention has 10^5 - preferably preferred $5x10^9$ ohm / sq. An elastic modulus of a magnetic layer which is extended 0.5% and comes out preferably a running direction and the cross direction 100-2000kg[/mm] ^{2}Breaking strength is preferred and an elastic modulus of 1 - 30 kg/cm^2 and a magnetic recording medium A running directionThe cross direction is preferred100-1500kg[/mm] ^{2} and remains mileage are preferredand a heat shrinkage rate in all temperature of 100 ** or less is 0.1% or less most preferably 0.5% or less still more preferably 1% or less 0.5% or less.
[0083]A residual solvent contained in a magnetic layer is below 10 mg/m^2 still more preferablyand its way with few residual solvents contained in a magnetic layer than a residual solvent contained in a non-magnetic layer is [below 100 mg/m^2] preferably preferred. Both voidage of below 30 capacity % that a magnetic layer and a non-magnetic layer have is below 10 capacity % still more preferably preferably. Although the one where voidage of a non-magnetic layer is larger than voidage of a magnetic layer is preferredit may be small as long as voidage of a non-magnetic layer is not less than 5%.
[0084]Although a magnetic recording medium of this invention has two or more layersaccording to the purposebeing presumed easily can change these physical properties on each class. For exampleit is making an elastic modulus of the upper layer of a magnetic layer lower than this lower layerand improving a hit to a head of a magnetic recording medium etc. at the same time it makes high a lower layer elastic modulus of a magnetic layer and raises running durability.
[0085]A magnetic layer applied on a base material by a wet-on-wet coating method dries a formed magnetic layerafter performing processing to which orientation of the ferromagnetic powder in a layer is carried out as occasion demands. Smooth surface-ized processing is performed as occasion demandsor it judges in desired shapeand a magnetic recording medium of this invention is manufactured. Although magnetic recording media of this invention may be tapessuch as a video use and an audio useor may be a floppy disk and a magnetic disk of a data recording useLack of a signal by generating of a dropout with high recording density is [as opposed to / especially / a disk like medium of a data recording use which becomes fatal] effective.
[0086]Namelyby using a magnetic recording medium of this invention as a magnetic recording diskThe overwrite characteristic that high-density magnetic recording is possible and it is indispensable to a digital data recording medium especially used for preservation and read-out in computer informationFor exampleit has the advantage that do not fall even if it becomes the high density recording of as [whose a shortest recording wavelength is 1.5 micrometers or less]and running durability does not falleither.
[0087]Not only when a recording wavelength carries out short wavelength formationbut when track density becomes highby using a magnetic recording disk of this inventionthere are few cross talks of a signal and record excellent in the separability of a peak shift can be performed. Thereforerecording track width is the conditions track density 14 track / more than 50 micrometers or less and mmeven if a shortest recording wavelength carries out record of 1.5 micrometers or lessit excels in overwrite fitnessand good record and reproduction are possible also for running durability.
[0088]
[Example]Hereafteran example explains this invention still more concretely. It being what can be changed in the ingredient shown here and the range in which an operational sequence does not deviate from the pneuma of this invention comparatively is understood easily for what is engaged in this industry. Thereforethis invention should not be restricted to the following example. It is "weight sections" that all it is with a "part."
[0089]The coating liquid for non-magnetic layersthe magnetic coating liquid for lower layers of a magnetic layerand the magnetic coating liquid for the upper layers of the magnetic layer were prepared by one or less-example formula.
[0090]
Coating liquid for non-magnetic layers Nonmagnetic inorganic powder 90 copies Granular TiO_2 (TY50 by Ishihara Sangyo KaishaLtd.)
mean particle diameter 0.34 micrometer Specific surface area by a BET adsorption method . 5.9 m^2/g pH 5.9. carbon black Ten copies Mean particle diameter 30mmicro. DBP oil absorption 350 ml / 100g pH. 9.5 Specific surface area by BET adsorption method 950m^2/g. volatile matter content 1.0% VCM/PVC system copolymer . Copies [14] -SO_3Na Group 1X10 $^{-4}$Eq/g Content Polyester Polyurethane Resin Five Copies Neopentyl Glycol / Caprolactone Polyol / MDI =0.9/2.6/1 -SO_3Na Group . 1x10 $^{-4}$eq/g content sec-butyl stearate Five copies Five copies of 2-butoxy-1-ethyl stearate Oleic acid One copy Methyl ethyl ketone 200 copies[0091]
The magnetic coating liquid ferromagnetism fine-metallic-powder end for lower layers of a magnetic layer 100 copies Presentation Fe/nickel=96/4 Hc 1500OeSpecific surface area by a BET adsorption method 58-m^2/g crystallite size 195 A Mean particle diameter (long axis length) 0.20 micrometeracicular ratio 10 saturation magnetization (sigma$_S$). 130 emu/g VCM/PVC system copolymer . Copies [14] -SO_3Na Group 1X10 $^{-4}$Eq/g Content Polyester Polyurethane Resin Three Copies Neopentyl Glycol / Caprolactone Polyol / MDI =0.9/2.6/1 -SO_3Na Group . 1x10 $^{-4}$eq/g content alpha alumina (mean-particle-diameter .) 0.3micrometer 2 copy Carbon black (mean particle diameter 0.10 micrometer) 0.5 copy sec-butyl stearate Five copies 2-butoxy-1-ethyl stearate Five copies Oleic acid One copy Methyl ethyl ketone 200 copies[0092]
Magnetic coating liquid for the upper layers of a magnetic layer The end of ferromagnetic fine metallic powder 100 copies Presentation Fe/nickel=97/3 Hc 1550OeSpecific surface area ^{2}[of 60 m]/g crystallite size by a BET adsorption method 160 A Mean particle diameter (long axis length) 0.16 micrometeracicular ratio 10 saturation magnetization (sigma$_S$). 127 emu/g VCM/PVC system copolymer . Copies [14] -SO_3Na Group 1X10 $^{-4}$Eq/g Content Polyester

Polyurethane Resin Three Copies Neopentyl Glycol / Caprolactone Polyol / MDI =0.9/2.6/1 -SO_3Na Group . 1x10^{-4}eq/g content alpha alumina (mean-particle-diameter .) 0.3micrometer 2 copy Carbon black (mean particle diameter 0.10 micrometer) 0.5 copy sec-butyl stearate Five copies 2-butoxy-1-ethyl stearate Five copies Oleic acid One copy Methyl ethyl ketone 200 copies[0093]After kneading each ingredient by a continuation kneaderit was made to distribute about each of the three above-mentioned paints using a sand mill. To the coating liquid for non-magnetic layerspolyisocyanate (Japanese polyurethane company makecoronate L) at the obtained dispersion liquid Ten copiesIt added 12 copies each to the object for the upper layers and the magnetic coating liquid for lower layers of the magnetic layer40 copies of butyl acetate was further added to eachit filtered using the filter which has a 1-micrometer average pore sizeand the object for the upper layers and the coating liquid for lower layers for the coating liquid for non-magnetic layer formation and magnetic layer formation were preparedrespectively.

[0094]So that the thickness after drying the obtained coating liquid for non-magnetic layer formation may be set to 2 micrometersSo that the thickness after desiccation of the lower layer of a magnetic layer may be set to 0.25 micrometer on it immediately after thatSo that the thickness after desiccation of the upper layer of a magnetic layer may furthermore be set to 0.20 micrometer on it immediately after thatMain average surface roughness performs simultaneous multistory spreading at 62 micrometers in thickness on the polyethylene terephthalate base material which is 0.01 micrometer using each coating liquid the lower layer of a magnetic layerand for the upper formationWhile each class is still in a damp or wet conditionthe frequency of 50 Hz and the magnetic field intensity of 200 gauss again The frequency of 50 HzIt is passed through the inside of two 120 gauss alternating current magnetic field generatorsand random orientation treatment is performedIt processes after desiccation with seven steps of calendar devices (linear pressure 300 kg/cmtemperature 90 **)After piercing to 3.5 inch sizes and performing surface polish processingthe liner put into the 3.5-inch cartridge installed insideand added the predetermined mechanism elementand a 3.5-inch floppy disk was obtained. .

[0095]The ferromagnetic metal powder used for the upper layer of the magnetic layer in example 2 Example 1 was changed as followsand also the sample was created like Example 1.

Ferromagnetic hexagonal ferrite powder Specific surface area by 100-copy Hc 1520Oe and a BET adsorption method 45m^2/g Mean particle diameter (plate diameter) 0.10 micrometertabular ratio Seven Saturation magnetization ($sigma_S$) 57 emu/g[0096]in example 3 Example 1the thickness after desiccation is set to 2 micrometers in the coating liquid for non-magnetic layers -- as -- immediately after that -- an it top -- the lower layer thickness of the magnetic layer was set to 0.40 micrometerand it was made for the upper thickness to be further set to 0.30 micrometer on it just behind thatand also the sample was created like Example 1.

[0097]in example 4 Example 2the thickness after desiccation is set to 2 micrometers in the coating liquid for non-magnetic layers -- as -- immediately after that -- an it top -- the lower layer thickness of the magnetic layer was set to 0.40 micrometerand it was made for the upper thickness to be further set to 0.30 micrometer on it just behind thatand also the sample was created like Example 1.

[0098]The ferromagnetic metal powder used for the upper layer of the magnetic layer in comparative example 1 Example 1 was changed as followsand also the sample was created like Example 1.

The end of ferromagnetic fine metallic powder 100 copies Presentation Specific surface area by Fe/nickel=97/3 Hc 1550Oeand a BET adsorption method 44-m^2/g crystallite size 250 A Mean particle diameter (long axis length) 0.27 micrometeracicular ratio 11 Saturation magnetization ($sigma_S$) 133 emu/g[0099]The ferromagnetic metal powder used for the upper layer of the magnetic layer in comparative example 2 Example 2 was changed as followsand also the sample was created like Example 2.

Ferromagnetic hexagonal ferrite powder 100 copies Specific surface area by Hc 1540Oe and a BET adsorption method 40m^2/g Mean particle diameter (plate diameter) 0.25 micrometertabular ratio Six Saturation magnetization ($sigma_S$) 59 emu/g[0100]in comparative example 3 Example 1the thickness after desiccation is set to 2 micrometers in the coating liquid for non-magnetic layers -- as -- immediately after that -- an it top -- the lower layer thickness of the magnetic layer was set to 0.60 micrometerand it was made for the upper thickness to be further set to 0.40 micrometer on it just behind thatand also the sample was created like Example 1.

[0101]The addition of the titanium oxide used for the coating liquid for non-magnetic layers in comparative example 4 Example 1 and carbon black was changed as followsand also the sample was created like Example 1.

100 copies of TiO_2 Carbon black Zero copy (with no addition)

[0102]A non-magnetic layer is not provided on the same polyethylene terephthalate base material as Example 1 by the object for the lower layers of the magnetic layer of comparative example 5 Example 1and the formula of each coating liquid for the upper layersThe lower layer thickness of the magnetic layer was set to 0.40 micrometerand it was made for the upper thickness to be further set to 0.30 micrometer on it immediately after thatand also the sample was created like Example 1.

[0103]Use only the coating liquid for the upper layers immediately after thatwithout providing the lower layer of a magnetic layer on itand it was made for the thickness of the magnetic layer to be set to 0.20 micrometer so that the thickness after desiccation may be set to 2 micrometers in the coating liquid for non-magnetic layers in comparative example 6 Example 1and also the sample was created like Example 1.

[0104]Use only the coating liquid for the upper layers immediately after thatwithout providing the lower layer of a magnetic layer on itand it was made for the thickness of the magnetic layer to be set to 0.20 micrometer so that the thickness after desiccation may be set to 2 micrometers in the coating liquid for non-magnetic layers in comparative

example 7 Example 2and also the sample was created like Example 2.
[0105]It dried after spreading so that it might become 2 micrometers of dry thickness tastes first on the polyethylene terephthalate base material as Example 1 only with same coating liquid for non-magnetic layers by the formula of each coating liquid of the non-magnetic layer of example 5 Example 1and a magnetic layer. Thenwhile it was in the after-spreading damp or wet condition so that the dry thickness taste might be set to 0.25 micrometer in the lower layer of a magnetic layer on the non-magnetic layerrandom orientationdesiccationand a calendar process were performed on the same conditions as Example 1. While it was in the after-spreading damp or wet condition so that the thick taste after furthermore drying the upper layer on the lower layer of this magnetic layer after that might be set to 0.20 micrometerrandom orientationdesiccationand a calendar process were performed on the same conditions as Example 1and the sample was created.
[0106]The kind of carbon black currently used for the non-magnetic layer in example 6 Example 1 was changed as followsand also the sample was created like Example 1.
Carbon black (#50 by a Mitsubishi carbon company) Ten copies Mean particle diameter 80mmicro DBP oil absorption Specific surface area by 63 ml / 100g pH 5.5 BET adsorption method 23m^2/g Volatile matter content Each sample of the floppy disk produced by doing in this way 1.0%It measured with the following valuation method.
[0107]Surface electric resistance (ohm/sq): The Takeda Riken make and TR-8611A (digital superinsulation ohm-meter) were usedand it measured by the method indicated to JISX6101.9.4.
Measurement of a reproducing output : Measurement of a reproducing output uses 0.30 micrometer of gap length's metal yne gap head using a disk test equipment SK606made from Tokyo engineering B typeAfter 2f recording frequency's having been 1250 kHz and recording 1f recording frequency in a position 24.6 mm in radius at 625 kHzrespectivelythe reproducing output of the head amplification machine was measured with the Tektronix oscilloscope 7633 type. The reproducing output set the output of Example 1 to 100and showed it with the relative value.
[0108]Overwrite : The overwrite characteristic is a position 39.5 mm in radius using the above-mentioned test equipmentAfter recording 312.5 kHz on an alternating-current-magnetic-conditioning finishing sample and measuring the output 01 of a 312.5-kHz ingredient (dB) by an ADVANTEST CORP. make TR4171 type spectrum analyzerOverwrite of the 1 MHz was carried out promptlyand it asked for the overwrite 02-01 (dB) from the output 02 of the 312.5-kHz ingredient at that time (dB).
[0109]Center-line average surface roughness (Ra): It measured by 0.25 mm of cutoff using the three-dimensional surface roughness meter (product made from the Kosaka research).
Running durability: After recording on all the 240 tracks with the recording frequency of 625 kHz using the floppy disk drive FD1331 type by NEC Corp.the radius carried out the thermostat cycle test which makes the thermostat cycle flow of a statement one cycle in a 37.25-mm position in Table 1 from the center. Under this thermostat conditionit had a run state at the time of making it run to 12 million times by passing timeand running durability was evaluated.
[0110]
[Table 1]

[0111]The evaluation result of each weighted solidity of the example and comparative example which were acquired with the above valuation method was shown in Table 2.
[0112]
[Table 2]

[0113]Examples 1-4 of this invention show a 1f reproducing outputa 2f reproducing outputand the characteristic outstanding in each magnetic parametric performance of over-writingand show the result stable also in surface electric resistance and cycle running durability. Both the comparative example 1 which uses the ferromagnetic metal powder of larger size than the mean particle diameter of the ferromagnetic metal powder used in the lower layer of the magnetic layer for the upper layersand the comparative example 2 which uses ferromagnetic hexagonal ferrite powder similarly were inferior in a 2f output.
[0114]Overwrite characteristic with what [sufficient] has a thick after-spreading dry thickness taste of a magnetic layer is not acquired. Usuallyin digital recording mediathe overwrite characteristic of -30 dB or less is needed (comparative example 3). The surface specific resistance value became high and the dropout generated both the comparative example 4 which did not provide a non-magnetic layerand the comparative example 5 which did not add carbon black of the non-magnetic layer in endurance for a short time.
[0115]As for both the comparative examples 6 and 7 made only into the upper layer of a magnetic layer without providing the lower layer of a magnetic layer on a non-magnetic layera 1f reproducing output declined. As for Example 5 which is not simultaneous multistory spreadingsurface nature and a reproducing output declined a little. In the DBP oil absorption of carbon blackin what has low still larger mean particle diametersurface nature and a reproducing output other than endurance are declining a little. (Example 6).
[0116]
[Effect of the Invention]While this invention has preferably the non-magnetic layer which distributed nonmagnetic powder in the binding material on the nonmagnetic substrateand this non-magnetic layer in a damp or wet conditionthe

lower layer of the magnetic layer which distributed ferromagnetic powder in the binding material is provided on itWhile this lower layer is furthermore in a damp or wet conditionare a magnetic recording medium which provides further the upper layer which distributed ferromagnetic powder in the binding material on itand the magnetic layer overall thickness of the lower layer of these magnetic layers and the upper layer of a magnetic layer at 0.8 micrometer or less. The ferromagnetic powder with which the ferromagnetic powder used for the lower layer of a magnetic layer is further used for the upper layer on it with ferromagnetic metal powder is the ferromagnetic metal powder or the ferromagnetic hexagonal ferrite powder below the mean particle diameter of the ferromagnetic metal powder used in a lower layer.
By making carbon black contain at least as nonmagnetic powder of further a lower layerit excels in running durabilitya surface electric resistance value can be made lowand the magnetic recording medium excellent in high density recording and running durability can be obtained.

WRITTEN AMENDMENT

[Written amendment]
[Filing date]May 13Heisei 5
[Amendment 1]
[Document to be Amended]Specification
[Item(s) to be Amended]0041
[Method of Amendment]Change
[Proposed Amendment]
[0041]This invention can use the thing of all processes as carbon black which can be used for a non-magnetic layer. For examplefurnace blackthermal blackacetylene blackchannel blacklamp blacketc. can be used. As a concrete example#3950B by Mitsubishi Kasei Corp.Ketchen black EC by lion AkzoKetchen black ECDJ-500Ketchen black ECDJ-600etc. are mentioned.
[Amendment 2]
[Document to be Amended]Specification
[Item(s) to be Amended]0112
[Method of Amendment]Change
[Proposed Amendment]
[0112]
[Table 2]

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(54)【発明の名称】　磁気記録媒体

(57)【要約】

【目的】　高再生出力、オーバーライト特性等の電磁変換特性が良好で、帯電しにくくかつ走行耐久性の優れた超高密度磁気記録媒体を提供すること。

【構成】　非磁性支持体上に非磁性粉末及び結合剤樹脂を主体とする非磁性層並びに強磁性粉末及び結合剤樹脂を主体とする磁性層が、この順で形成されてなる磁気記録媒体において、前記磁性層は、上層と下層の少なくとも２層からなり、前記下層の強磁性粉末の平均粒径は前記上層の強磁性粉末の平均粒径よりも大きく、該磁性層の全厚は０．８μm以下であることを特徴とする磁気記録媒体であり、好ましくは、該非磁性層及び磁性層は、ウエット・オン・ウエット塗布方式で形成されたもので、強磁性粉末として、強磁性金属粉末または強磁性六方晶系フェライト粉末を使用し、非磁性粉末として少なくともカーボンブラックを使用する。

【特許請求の範囲】

【請求項１】　非磁性支持体上に非磁性粉末及び結合剤樹脂を主体とする非磁性層並びに強磁性粉末及び結合剤樹脂を主体とする磁性層が、この順で形成されてなる磁気記録媒体において、前記磁性層は、上層と下層の少なくとも２層からなり、前記下層の強磁性粉末の平均粒径は前記上層の強磁性粉末の平均粒径よりも大きく、該磁性層の全厚は０．８μｍ以下であることを特徴とする磁気記録媒体。

【請求項２】　前記非磁性層及び磁性層は、ウエット・オン・ウエット塗布方式で形成されたものであることを特徴とする請求項１記載の磁気記録媒体。

【請求項３】　前記強磁性粉末が強磁性金属粉末である請求項１または請求項２記載の磁気記録媒体。

【請求項４】　前記強磁性粉末が強磁性六方晶系フェライト粉末である請求項１～請求項３のいずれか１項に記載の磁気記録媒体。

【請求項５】　前記非磁性粉末の少なくとも一部がカーボンブラックである請求項１～請求項４のいずれか１項に記載の磁気記録媒体。

【請求項６】　前記カーボンブラックは、平均粒径が４０mμ以下であり、かつＤＢＰ吸油量が３００mＬ／１００ｇ以上である請求項１～請求項５のいずれか１項に記載の磁気記録媒体。

【請求項７】　前記磁気記録媒体が磁気記録ディスクである請求項１～請求項６のいずれか１項に記載の磁気記録媒体。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、高密度記録用磁気記録媒体に関し、特に、データ記録用の磁気ディスクに適した磁気記録媒体に関するものである。

【０００２】

【従来の技術】磁気記録技術は、媒体の繰り返し使用が可能であること、信号の電子化が容易であり周辺電子機器との組み合わせによるシステムの構築が可能であること、信号の修正も簡単にできること等の他の記録方式にはない優れた特長を有することから、ビデオ、オーディオ、コンピューター用途等を始めとして様々な分野で幅広く利用されてきた。そして、機器の小型化、記録再生信号の高品位化、記録の長時間化、記録容量の増大等の要求に対応するために、記録媒体に対しては記録密度のより一層の向上が常に望まれてきた。そして、塗布型の磁気記録媒体においては、強磁性粉末の粒子サイズを小さくしたり、その分散性を向上させたり、その磁性層中での充填度を高めたりする方策が種々提案されているさらに有効な手段として、磁気特性の優れた強磁性金属粉末や六方晶フェライトなどを用いることも行われている。

【０００３】また、ＯＡ機器としてのミニコン、パソコンの普及にともない外部記憶媒体としての磁気記録ディスクの普及が著しく、磁気記録ディスクの使用頻度が広がって、温度湿度に関し、幅広い環境条件で使用・保存され、また使用環境の塵埃も多い場所で使用されるようになってきた。特に、記録の大容量化、小型化を達成するために記録密度の向上が強く要望されているが、従来のような針状強磁性粉末を用いて高密度記録に適する磁気記録媒体を得るには、針状強磁性粉末の最大寸法を記録波長、あるいは記録ビット長よりも十分小さくする必要がある。現在、針状強磁性粉末として０．３μｍ程度の寸法のものがすでに実用に供されており、最短記録波長約１μｍ以下が可能になっている。

【０００４】今後さらに高密度の記録が可能な媒体を得るには、針状強磁性粉末の寸法をなお一層小さくする必要がある。しかしそのような小さな針状強磁性粉末においては、太さが１００Å以下と極めて細くなり、粒子体積としても10^{-17} ｃm^3 以下と極めて小さくなるため、熱擾乱、表面の効果によって磁気特性が低下し、又磁性塗膜に磁界を加えても十分な配向が得られない等の問題がある。

【０００５】高密度記録に対応する強磁性粉末として、これまで強磁性金属粉末が検討されてきており、また近年平板状で板面に垂直な方向に磁化容易軸を有する六方晶系フェライト粒子を強磁性粉末として用いる磁気記録媒体が開発された（例えば、特開昭５８－６５２５号、同５８－６５２６号等）。これらによって、強磁性粉末の平均粒径は０．０５μｍ以下が可能となり、高密度記録化が可能となった。

【０００６】さらに高密度記録のため大幅な狭トラック幅が要求されてきている。これらの要求を満たすため磁気ディスクにおいても高出力や高記録密度が期待できる強磁性金属粉末や強磁性六方晶系フェライト粉末の使用が検討されており、サイズの小型化や媒体の記録密度の向上の要求に応えるべく鋭意開発、実用化が検討されている。特に高記録密度およびオーバーライト電磁変換特性の向上として磁性層の薄層化、高出力が望まれ、薄層化に伴い走行耐久性の大幅劣化が懸念されている。

【０００７】即ち、通常、フロッピーディスク等のコンピューター用途の磁気記録媒体においては、磁気波長の異なる記録信号の重ね書き（オーバーライト）が必要であるが、従来は、周波数で２倍の関係にある２種類の信号、１ｆ及び２ｆ信号のオーバーライトができれば良かったが、最近強く要望されている１０Ｍバイト以上の高容量の磁気記録ディスクに対しては、記録波長が短くなっただけではなく、ＲＬＬ信号などの周波数比３：８のより広帯域にある複数の信号のオーバーライトが要求されている。記録波長が短く、記録周波数の差が大きい信号を使用した場合、記録波長の短い信号を記録波長が長い信号の上に重ね書き（オーバーライト）をうまく行うためには、特開昭５８－１２２６２３号公報、特開昭６

１－７４１３７号公報等に開示されているように、単に磁性層の磁気特性を向上させるだけでは限界があった。

【０００８】すなわち、今までの１．０μm以上の厚さの磁性層では、先に記録されているより長い波長の記録信号の上により短い記録信号を重ね書きしても磁力線が磁性層の深いところまで達しないために、先に記録されたより長い波長の信号が消去できないのである。また、記録密度の向上にともない記録ヘッドのギャップが狭くなってきている。これにともない、媒体の厚味方向への十分な記録が困難になってきている。

【０００９】そこで、上記問題を解消するために磁性層を１μm以下に薄くすると磁性層は剥離し易くなり、ドロップアウトの発生要因となる等走行耐久性が確保できず、信頼性が低下するという問題が起こった。従って、前記の高密度記録化に対応し得る磁気記録媒体を提供するためには、特に再生出力の向上およびオーバーライト特性の確保および走行耐久性が大きな問題となってきた。

【００１０】また、磁気記録媒体の走行時の帯電は、塵埃の付着によるドロップアウト数の増大を招き、特にデータ記録用の磁気記録媒体の場合は、それによるエラー・レートが致命的な欠陥となった。この帯電の問題を改良するために、磁性層中に帯電を防止するために添加物を加える方法が通常取られており、中でもカーボンブラックを添加する方法が最も有効であり、広く採用されている。しかしながら、前記の高密度記録用の磁気記録媒体にあっては、カーボンブラックの添加は、磁性体充填度を下げる出力の低下を招くので、その添加量に限界があり、帯電防止への充分な対処ができなかった。

【００１１】特に、前記の強磁性六方晶系フェライト粉末は、Ｃｏ－Fe_2O_3 強磁性粉末、強磁性金属粉末等と比較し、飽和磁化量が低く、高い出力が得られ難いため高出力の磁気記録媒体を提供するには充填密度を上げなければならないが、微粒子でありかつ形状が六方晶形であるがため、分散性が従来の強磁性粉末に比較して劣り、帯電防止性、高再生出力を確保することが基本的に困難である。

【００１２】前記の帯電の防止、高出力化と耐久性の向上を満足させるための種々提案が開示されている。（特開昭５５－５５４３１号、特開昭５５－５５４３２号、特開昭５５－５５４３３号、特開昭５５－５５４３４号、特開昭６０－１６４９２６号、特開昭５５－５５４３６号、特開昭６２－３８５２３号、特開昭６２－１５９３３７号公報等）即ち、磁性層と支持体の間に中間層を設けるものであるが、中間層にカーボンブラックと結合剤樹脂を塗布し、その後その上に磁性層を形成しようとするものである。

【００１３】しかしながら、この方法は、走行耐久性を改善するめには有効であったが、高密度記録の磁気記録媒体であって、十分な走行耐久性を確保しつつ、しかも、優れた電磁変換特性、すなわち高再生出力、オーバーライト特性を満足することはできなかった。そして、この問題に対処する有効な方法はいまだに提案されていない。

【００１４】

【発明が解決しようとする課題】本発明は、前記従来技術の問題点に鑑みなされたものであって、高再生出力、オーバーライト特性等の電磁変換特性が良好で、帯電しにくくかつ走行耐久性の優れた超高密度磁気記録媒体を提供することを目的としている。

【００１５】

【課題を解決するための手段】本発明は、非磁性支持体上に非磁性粉末及び結合剤樹脂を主体とする非磁性層並びに強磁性粉末及び結合剤樹脂を主体とする磁性層が、この順で形成されてなる磁気記録媒体において、前記磁性層は、上層と下層の少なくとも２層からなり、前記下層の強磁性粉末の平均粒径は前記上層の強磁性粉末の平均粒径よりも大きく、該磁性層の全厚は０．８μm以下であることを特徴とする磁気記録媒体であり、これにより上記課題を解決できる。

【００１６】本発明は、非磁性支持体の上に非磁性層及び磁性層をこの順に設けた磁気記録媒体であって、かつ磁性層が２層以上の複層構造であり、その磁性層の全厚を０．８μm以下に限定したことを特徴とする。また、第２に磁性層を複層構造とすると共に下層の強磁性粉末の平均粒径を上層のそれより大きくしたことを特徴とする。

【００１７】ところで、一般に平均粒径が小さい強磁性粉末は、長波長の記録波長域では出力は低いが、短波長領域での出力は高く、平均粒径が大きい粒子はその逆であることが知られている。また、長波長の記録信号は、磁性層の深いところまで有効であることが知られている。本発明では、平均粒径の違いによる強磁性粉末の周波数特性が磁性層の下層と上層でそれぞれ特徴的な上記性質が発揮されるように構成したものである。

【００１８】即ち、本発明は、磁性層の厚さを薄くすることにより、自己減磁損失を低減して再生出力を向上したと共に重ね書き特性、例えば、前に記録された長波長の信号の上に後から短波長の信号を記録しても前者の信号の記録磁化の影響を受けずに後者の信号が有効に記録される特性を改善したものである。また、本発明は、磁性層及び非磁性層を形成するための塗布方式は特に制限はないが、好ましくは、非磁性層と磁性層をウエット・オン・ウエット塗布方式で形成すると、厚味が均一で表面が平滑しかも非磁性層との密着性が良好な磁性層が形成できるため、再生出力の高い、耐久性の優れた磁気記録媒体を提供する。

【００１９】磁性層を非磁性層を乾燥してから塗布して形成することもできる。しかし、磁性層の表面性を確保しにくく、また、極端な場合には、磁性層にピンホール

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(54)【発明の名称】　磁気記録媒体

(57)【要約】

【目的】　高再生出力、オーバーライト特性等の電磁変換特性が良好で、帯電しにくくかつ走行耐久性の優れた超高密度磁気記録媒体を提供すること。

【構成】　非磁性支持体上に非磁性粉末及び結合剤樹脂を主体とする非磁性層並びに強磁性粉末及び結合剤樹脂を主体とする磁性層が、この順で形成されてなる磁気記録媒体において、前記磁性層は、上層と下層の少なくとも２層からなり、前記下層の強磁性粉末の平均粒径は前記上層の強磁性粉末の平均粒径よりも大きく、該磁性層の全厚は０．８μｍ以下であることを特徴とする磁気記録媒体であり、好ましくは、該非磁性層及び磁性層は、ウエット・オン・ウエット塗布方式で形成されたもので、強磁性粉末として、強磁性金属粉末または強磁性六方晶系フェライト粉末を使用し、非磁性粉末として少なくともカーボンブラックを使用する。

【特許請求の範囲】

【請求項１】　非磁性支持体上に非磁性粉末及び結合剤樹脂を主体とする非磁性層並びに強磁性粉末及び結合剤樹脂を主体とする磁性層が、この順で形成されてなる磁気記録媒体において、前記磁性層は、上層と下層の少なくとも２層からなり、前記下層の強磁性粉末の平均粒径は前記上層の強磁性粉末の平均粒径よりも大きく、該磁性層の全厚は０．８μm以下であることを特徴とする磁気記録媒体。

【請求項２】　前記非磁性層及び磁性層は、ウエット・オン・ウエット塗布方式で形成されたものであることを特徴とする請求項１記載の磁気記録媒体。

【請求項３】　前記強磁性粉末が強磁性金属粉末である請求項１または請求項２記載の磁気記録媒体。

【請求項４】　前記強磁性粉末が強磁性六方晶系フェライト粉末である請求項１～請求項３のいずれか１項に記載の磁気記録媒体。

【請求項５】　前記非磁性粉末の少なくとも一部がカーボンブラックである請求項１～請求項４のいずれか１項に記載の磁気記録媒体。

【請求項６】　前記カーボンブラックは、平均粒径が４０mμ以下であり、かつＤＢＰ吸油量が３００mL／１００g以上である請求項１～請求項５のいずれか１項に記載の磁気記録媒体。

【請求項７】　前記磁気記録媒体が磁気記録ディスクである請求項１～請求項６のいずれか１項に記載の磁気記録媒体。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、高密度記録用磁気記録媒体に関し、特に、データ記録用の磁気ディスクに適した磁気記録媒体に関するものである。

【０００２】

【従来の技術】磁気記録技術は、媒体の繰り返し使用が可能であること、信号の電子化が容易であり周辺電子機器との組み合わせによるシステムの構築が可能であること、信号の修正も簡単にできること等の他の記録方式にはない優れた特長を有することから、ビデオ、オーディオ、コンピューター用途等を始めとして様々な分野で幅広く利用されてきた。そして、機器の小型化、記録再生信号の高品位化、記録の長時間化、記録容量の増大等の要求に対応するために、記録媒体に対しては記録密度のより一層の向上が常に望まれてきた。そして、塗布型の磁気記録媒体においては、強磁性粉末の粒子サイズを小さくしたり、その分散性を向上させたり、その磁性層中での充填度を高めたりする方策が種々提案されているさらに有効な手段として、磁気特性の優れた強磁性金属粉末や六方晶フェライトなどを用いることも行われている。

【０００３】また、ＯＡ機器としてのミニコン、パソコンの普及にともない外部記憶媒体としての磁気記録ディスクの普及が著しく、磁気記録ディスクの使用頻度が広がって、温湿度に関し、幅広い環境条件で使用・保存され、また使用環境の塵埃も多い場所で使用されるようになってきた。特に、記録の大容量化、小型化を達成するために記録密度の向上が強く要望されているが、従来のような針状強磁性粉末を用いて高密度記録に適する磁気記録媒体を得るには、針状強磁性粉末の最大寸法を記録波長、あるいは記録ビット長よりも十分小さくする必要がある。現在、針状強磁性粉末として０．３μm程度の寸法のものがすでに実用に供されており、最短記録波長約１μm以下が可能になっている。

【０００４】今後さらに高密度の記録が可能な媒体を得るには、針状強磁性粉末の寸法をなお一層小さくする必要がある。しかしそのような小さな針状強磁性粉末においては、太さが１００Å以下と極めて細くなり、粒子体積としても10^{-17} cm^3 以下と極めて小さくなるため、熱擾乱、表面の効果によって磁気特性が低下し、又磁性塗膜に磁界を加えても十分な配向が得られない等の問題がある。

【０００５】高密度記録に対応する強磁性粉末として、これまで強磁性金属粉末が検討されてきており、また近年平板状で板面に垂直な方向に磁化容易軸を有する六方晶系フェライト粒子を強磁性粉末として用いる磁気記録媒体が開発された（例えば、特開昭５８－６５２５号、同５８－６５２６号等）。これらによって、強磁性粉末の平均粒径は０．０５μm以下が可能となり、高密度記録化が可能となった。

【０００６】さらに高密度記録のため大幅な狭トラック幅が要求されてきている。これらの要求を満たすため磁気ディスクにおいても高出力や高記録密度が期待できる強磁性金属粉末や強磁性六方晶系フェライト粉末の使用が検討されており、サイズの小型化や媒体の記録密度の向上の要求に応えるべく鋭意開発、実用化が検討されている。特に高記録密度およびオーバーライト電磁変換特性の向上として磁性層の薄層化、高出力が望まれ、薄層化に伴い走行耐久性の大幅劣化が懸念されている。

【０００７】即ち、通常、フロッピーディスク等のコンピューター用途の磁気記録媒体においては、磁気波長の異なる記録信号の重ね書き（オーバーライト）が必要であるが、従来は、周波数で２倍の関係にある２種類の信号、１ｆ及び２ｆ信号のオーバーライトができれば良かったが、最近強く要望されている１０Mバイト以上の高容量の磁気記録ディスクに対しては、記録波長が短くなっただけではなく、ＲＬＬ信号などの周波数比３：８のより広帯域にある複数の信号のオーバーライトが要求されている。記録波長が短く、記録周波数の差が大きい信号を使用した場合、記録波長の短い信号を記録波長が長い信号の上に重ね書き（オーバーライト）をうまく行うためには、特開昭５８－１２２６２３号公報、特開昭６

１－７４１３７号公報等に開示されているように、単に磁性層の磁気特性を向上させるだけでは限界があった。

【０００８】すなわち、今までの１．０μm以上の厚さの磁性層では、先に記録されているより長い波長の記録信号の上により短い記録信号を重ね書きしても磁力線が磁性層の深いところまで達しないために、先に記録されたより長い波長の信号が消去できないのである。また、記録密度の向上にともない記録ヘッドのギャップが狭くなってきている。これにともない、媒体の厚味方向への十分な記録が困難になってきている。

【０００９】そこで、上記問題を解消するために磁性層を１μm以下に薄くすると磁性層は剥離し易くなり、ドロップアウトの発生要因となる等走行耐久性が確保できず、信頼性が低下するという問題が起こった。従って、前記の高密度記録化に対応し得る磁気記録媒体を提供するためには、特に再生出力の向上およびオーバーライト特性の確保および走行耐久性が大きな問題となってきた。

【００１０】また、磁気記録媒体の走行時の帯電は、塵埃の付着によるドロップアウト数の増大を招き、特にデータ記録用の磁気記録媒体の場合は、それによるエラー・レートが致命的な欠陥となった。この帯電の問題を改良するために、磁性層中に帯電を防止するために添加物を加える方法が通常取られており、中でもカーボンブラックを添加する方法が最も有効であり、広く採用されている。しかしながら、前記の高密度記録用の磁気記録媒体にあっては、カーボンブラックの添加は、磁性体充填度を下げる出力の低下を招くので、その添加量に限界があり、帯電防止への充分な対処ができなかった。

【００１１】特に、前記の強磁性六方晶系フェライト粉末は、Ｃｏ－Fe_2O_3 強磁性粉末、強磁性金属粉末等と比較し、飽和磁化量が低く、高い出力が得られ難いため高出力の磁気記録媒体を提供するには充填密度を上げなければならないが、微粒子でありかつ形状が六方晶形であるがため、分散性が従来の強磁性粉末に比較して劣り、帯電防止性、高再生出力を確保することが基本的に困難である。

【００１２】前記の帯電の防止、高出力化と耐久性の向上を満足させるための種々提案が開示されている。（特開昭５５－５５４３１号、特開昭５５－５５４３２号、特開昭５５－５５４３３号、特開昭５５－５５４３４号、特開昭６０－１６４９２６号、特開昭５５－５５４３６号、特開昭６２－３８５２３号、特開昭６２－１５９３３７号公報等）即ち、磁性層と支持体の間に中間層を設けるものであるが、中間層にカーボンブラックと結合剤樹脂を塗布し、その後その上に磁性層を形成しようとするものである。

【００１３】しかしながら、この方法は、走行耐久性を改善するめには有効であったが、高密度記録の磁気記録媒体であって、十分な走行耐久性を確保しつつ、しかも、優れた電磁変換特性、すなわち高再生出力、オーバーライト特性を満足することはできなかった。そして、この問題に対処する有効な方法はいまだに提案されていない。

【００１４】

【発明が解決しようとする課題】本発明は、前記従来技術の問題点に鑑みなされたものであって、高再生出力、オーバーライト特性等の電磁変換特性が良好で、帯電しにくくかつ走行耐久性の優れた超高密度磁気記録媒体を提供することを目的としている。

【００１５】

【課題を解決するための手段】本発明は、非磁性支持体上に非磁性粉末及び結合剤樹脂を主体とする非磁性層並びに強磁性粉末及び結合剤樹脂を主体とする磁性層が、この順で形成されてなる磁気記録媒体において、前記磁性層は、上層と下層の少なくとも２層からなり、前記下層の強磁性粉末の平均粒径は前記上層の強磁性粉末の平均粒径よりも大きく、該磁性層の全厚は０．８μm以下であることを特徴とする磁気記録媒体であり、これにより上記課題を解決できる。

【００１６】本発明は、非磁性支持体の上に非磁性層及び磁性層をこの順に設けた磁気記録媒体であって、かつ磁性層が２層以上の複層構造であり、その磁性層の全厚を０．８μm以下に限定したことを特徴とする。また、第２に磁性層を複層構造とすると共に下層の強磁性粉末の平均粒径を上層のそれより大きくしたことを特徴とする。

【００１７】ところで、一般に平均粒径が小さい強磁性粉末は、長波長の記録波長域では出力は低いが、短波長領域での出力は高く、平均粒径が大きい粒子はその逆であることが知られている。また、長波長の記録信号は、磁性層の深いところまで有効であることが知られている。本発明では、平均粒径の違いによる強磁性粉末の周波数特性が磁性層の下層と上層でそれぞれ特徴的な上記性質が発揮されるように構成したものである。

【００１８】即ち、本発明は、磁性層の厚さを薄くすることにより、自己減磁損失を低減して再生出力を向上したと共に重ね書き特性、例えば、前に記録された長波長の信号の上に後から短波長の信号を記録しても前者の信号の記録磁化の影響を受けずに後者の信号が有効に記録される特性を改善したものである。また、本発明は、磁性層及び非磁性層を形成するための塗布方式は特に制限はないが、好ましくは、非磁性層と磁性層をウエット・オン・ウエット塗布方式で形成すると、厚味が均一で表面が平滑しかも非磁性層との密着性が良好な磁性層が形成できるため、再生出力の高い、耐久性の優れた磁気記録媒体を提供する。

【００１９】磁性層を非磁性層を乾燥してから塗布して形成することもできる。しかし、磁性層の表面性を確保しにくく、また、極端な場合には、磁性層にピンホール